BRL-MR-3982

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MEMORANDUM REPORT BRL-MR-3982

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PREPARATION OF THE HAN-BASED LIQUID PROPELLANTS FOR FIELD USE

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Form Approved
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1. AGENCY USE ONLY (Leave blank)	2. REPORT DATE	3. REPORT TYPE AND DATES COVERED
	June 1992	Final, Aug 89 - May 91

4. TITLE AND SUBTITLE

Preparation of the HAN-Based Liquid Propellants for Field Use

5. FUNDING NUMBERS

PR: 2FVTYA
WU: A1E1C1A1B

6. AUTHOR(S)

Nathan Klein and Charles S. Leveritt

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

U.S. Army Ballistic Research Laboratory
ATTN: SLCBR-DD-T
Aberdeen Proving Ground, MD 21005-5066

10. SPONSORING/MONITORING AGENCY REPORT NUMBER

BRL-MR-3982

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION/AVAILABILITY STATEMENT

Approved for public release; distribution unlimited

12b. DISTRIBUTION CODE

13. ABSTRACT *(Maximum 200 words)*

The propellant used in the 155-mm liquid propellant howitzer, LGP1846, is a colorless, odorless, homogeneous mixture of the salts hydroxylammonium nitrate (HAN) and triethanolammonium nitrate (TEAN) in aqueous solution. The lack of both color and odor could cause confusion and possible misuse, and an investigation of additives that will provide color and odor to the mixture was undertaken. The presence of HAN and TEAN in LGP1846 causes the mixture to be acidic. The dye to be used as a colorant must be water soluble, intensely colored in acidic media, and chemically stable in the presence of both oxidizing and reducing ionic species. The thiazine dyes, examples of which are Thionine Blue, Methylene Green, and the well known Methylene Blue, are probably the best candidates. The color range of these dyes is violet to green.

The purpose of an odorant in the colored LGP1846 is to alert operational personnel that a spill has occurred. Replacement of about 1% of the TEAN in the mixture with trimethylammonium nitrate (TMAN) would generate a distinctive, amine-like odor if the mixture came in contact with nonacidic substances such as skin, clothing, the ground, or the surface of a vehicle.

14. SUBJECT TERMS

hydroxylammonium nitrate, triethanolammonium nitrate, HAN, TEAM, dyes, methylene blue, odorants, trimethylammonium nitrate, TMAN, LGP1846, XM46

15. NUMBER OF PAGES

17

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
UNCLASSIFIED	UNCLASSIFIED	UNCLASSIFIED	SAR

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1. INTRODUCTION

The propellant LGP1846 is used in the 155-mm liquid propellant gun (LPG). It is an aqueous, stoichiometric mixture of the salts hydroxylammonium nitrate (HAN) and triethanolammonium nitrate (TEAN). The HAN:TEAN weight ratio is 3.17, water content is 20 weight-percent, and the mixture is colorless and odorless. Since both HAN and TEAN are the salts of weak bases and strong acids, the propellant is acidic and both oxidizing and reducing ions are available to react with any material added to the mixture. The highly desirable physical and chemical properties of the propellant (Decker et al. 1987; Klein, to be published) are the result of its composition and any modification should be approached cautiously because a seemingly innocuous change in composition could compromise one or more of these properties.

Factor 3.5.9, Recognition, of the Cannon Artillery Propulsion Evaluation Plan (CAPEP), calls for the propellant to have a distinctive color and odor in order to facilitate identification by field personnel. The colorless and odorless LGP1846 must, therefore, be modified in order to satisfy this requirement. This report addresses the rationale used in selecting potential colorants and odorants, and presents some of the data obtained with suitably modified propellant.

2. EXPERIMENTAL

It was assumed from the outset that the propellant was to remain homogeneous, since this property permits pumping of propellant charges into the gun without concern about uniformity of composition. The concept of a single additive that would provide both color and odor to the LGP1846 mixture was considered impractical, and as a result, each additive is addressed separately.

2.1 Color. The water miscibility, acidity, and reactivity of the propellant limits selection of a potential colorant to one that is water soluble, is readily visible in acidic media, and is not easily oxidized or reduced. These requirements reduce the myriad, water-soluble dyes to a relatively small group, because the acidic form of many of the dyes produce colors in the colorless-to-yellow range. Of the compounds remaining, the triphenylmethane dyes, such as Methyl Violet, Malachite Green, and Crystal Violet are readily oxidized and propellant samples colored with these dyes fade to colorless in approximately a week. The thiazine dyes are probably the best candidates for a propellant colorant (Lubs 1955). A general structural formula for these dyes is shown in Figure 1.

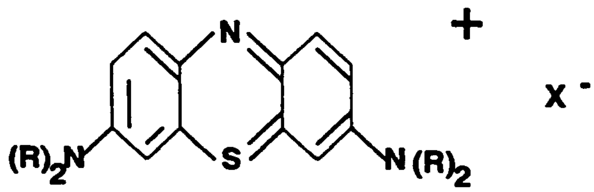

Figure 1. The Thiazine Dyes.

The specific color obtained is dependent on the functional groups, shown as R in Figure 1. The anion X does not affect color significantly, but can influence the solubility of the compound. The first of the thiazine dyes was Lauth's Violet in which R is hydrogen. Methylene Blue, in which R is CH_3 and X is chloride, was prepared in 1877 (German Patent 1886). Other examples of thiazine dyes are Thionine Blue, Methylene Green, and Methylene Violet (although the best known and most widely used is Methylene Blue). The color range of the various dyes in acidic solution is violet to green, and they are not easily oxidized or reduced. The absorption spectrum of Methylene Blue dissolved in propellant is shown in Figure 2. Molar extinction coefficient at 665 nm is 50,119 M^{-1} cm^{-1} so that a propellant sample 1 cm thick containing 10 ppm of dye will absorb 99.3% of the incident light at this wavelength.

Of greater practical interest than the absorption spectrum is how the propellant containing the dye appears to the eye. Figure 3 shows 5-mm-thick samples of LGP 1846, containing 10, 30, and 100 ppm Methylene Blue.

Since commercially available Methylene Blue is the chloride salt, it should be converted to nitrate because the presence of chloride, even in small quantities, inhibits propellant combustion (Klein and Sassé 1980). The nitrate salt was prepared by reaction of "certified" grade Methylene Blue (Aldrich 86,124-3)[1] with a stoichiometric quantity of aqueous silver nitrate. The resultant AgCl precipitate was removed by filtration, and the Methylene Blue solution concentrated at 100° C until the nitrate salt crystallized. The green-brown iridescent crystals are stable, and quite soluble. As seen in Figure 3, a rich, blue color is produced that is pale, but distinctly visible at 10 ppm of dye. It would seem doubtful that a color as deep as that obtained at 30 ppm would be needed. All of the samples shown in Figure 3 appear black when viewed in red light.

[1] Aldrich Chemical Company, Milwaukee, WI

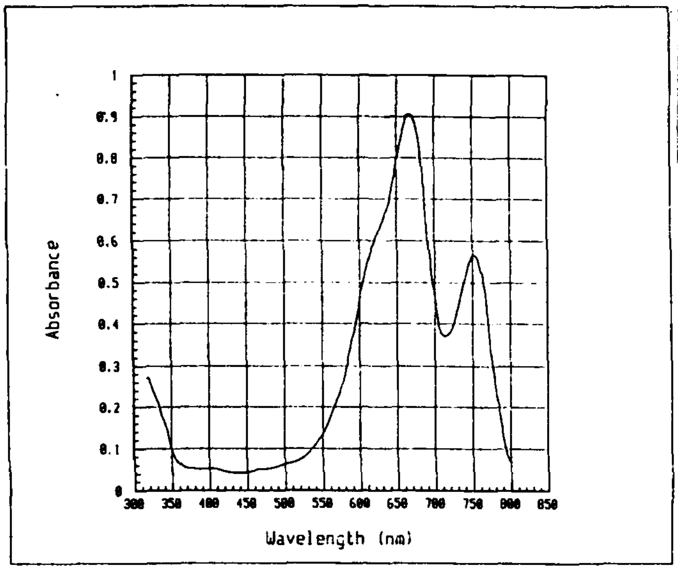

Figure 2. The Absorption Spectrum of Methylene Blue in LGP1846.

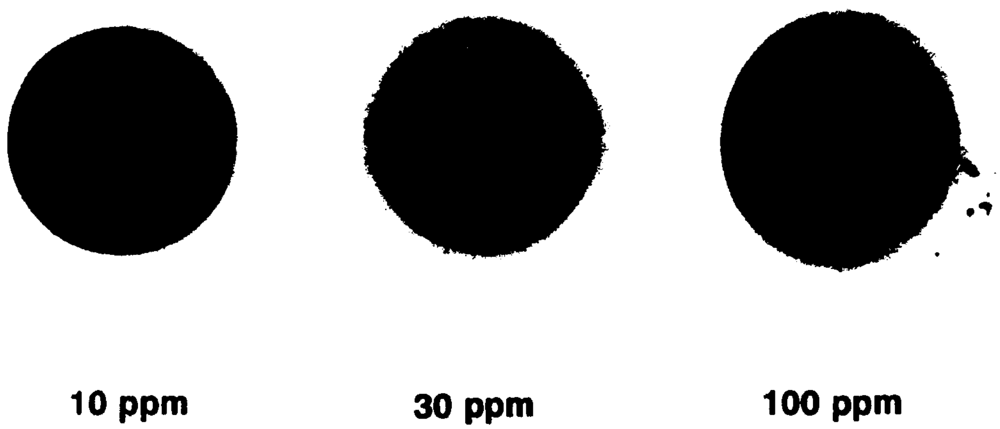

Figure 3. LGP1846 Containing 10, 30, and 100 ppm Methylene Blue.

2.2 Odor. It is expected that the propellant will be shipped and stored in distinctive, readily identifiable containers. If the LGP1846 is colored, the purpose of an odorant is not to identify the propellant, but rather to alert operational personnel that the material has been spilled. The odor should be well removed from those commonly encountered and quite possibly unpleasant. The addition of an intensely odoriferous compound to the propellant mixture would satisfy the requirement, but a second approach, in which an odor is created only when the material comes in contact with skin, the ground, or the vehicles in which it is transported or used, should also be considered.

Selection of a stable, propellant soluble, highly malodorous additive presents the same problems that were addressed in selecting a colorant. Aldehydes, ketones, and esters are not stable in LGP1846, and amines will be converted to their odorless ammonium ions. Organic sulfur compounds might be suitable, and a potential odorant is 2-mercaptoethanol, HS-CH_2CH_2-OH. The compound is reasonably water soluble, and possesses a sufficiently unpleasant odor to be readily identified. Although both the SH and OH groups should be susceptible to oxidation, preliminary tests indicate that LGP1846 saturated with 2-mercaptoethanol retains its odor after 2 years storage at room temperature. The specific concentration of 2-mercaptoethanol needed to produce an acceptable odor level is not known, but is expected to be higher than the dye concentrations discussed earlier. The addition of parts per thousand of sulfur to the propellant mixture could affect performance and could cause deterioration of gun components. If an odorant of this type was to be seriously considered, storage stability at elevated temperatures and deleterious effects on performance and/or gun components would have to be determined.

The second approach suggested, although seemingly more complex, is in fact, more direct and poses few potential problems. Replacement of 1% of the TEAN in LGP1846 by trimethylammonium nitrate (TMAN), the nitrate salt of trimethylamine, results in a mixture of composition 60.81% HAN, 18.19% TEAN, 1.00% TMAN, and 20% water. TMAN is stable and acts as a fuel during combustion in essentially the same manner as TEAN. In fact, TMAN was the fuel component of the propellant LGP1776, a precursor of LGP1846. The LGP1776 mixture was replaced with LGP1846 because the extensive hydrogen-bonding induced by the presence of TEAN increases density and improves low temperature properties. Thermochemical calculations indicate that the performance of the modified LGP1846 mixture differs from that of LGP1846 by about 0.3%. TMAN and the proposed mixture is odorless, but trimethylamine has a strong, unpleasant odor and is detected in the parts per billion range. The acidity of the propellant is comparable to that of lemon juice or vinegar and is reduced if the mixture comes in contact with common surfaces, such as skin, clothing, the ground, or the walls or floor of a vehicle. Under such conditions, the dissociation of TMAN will shift from the trimethylammonium ion toward trimethylamine, thereby creating the odor. Replacement of 1% of the TEAN with TMAN is not expected to cause significant deterioration of the physical properties of the mixture.

The presence of an odorant, either a permanent one or one produced by spillage, could create an essentially self-defeating situation. Olfactory sensitivity to compounds of the type suggested is extremely high and they will be detected if their concentration in air is in the parts per billion range. Once released, the odor may be extremely difficult to remove. As a result, the area around the gun might acquire a permanent odor, making it virtually impossible to detect a fresh spill.

3. CONCLUSIONS AND RECOMMENDATIONS

The presence of both color and odor in the propellant helps identify the material and alerts personnel to spills or other inadvertent release. In doing so, the problem of color and odor removal is also created. Color will be diluted and removed by washing with water, a recommended method of propellant disposal, but this technique will not necessarily remove the odorant because olfactory sensitivity is so very high. An odorant such as TMAN could be neutralized by washing with citric, acetic, or some other weak acid, but such cleaning procedures will add complexity and could cause damage to components.

If propellant is to be modified by addition of colorant and/or odorant, the newly formulated mixture will require complete evaluation with regard both to stability and efficacy. Although it is probable that the addition of Methylene Blue to achieve a dye concentration of about 10 ppm will have no significant effect on either the storage stability or performance of the propellant, assurance cannot be given at this time that this is the case. The use scenario for odorant containing propellant mixtures will require evaluation before more definitive plans can be formulated.

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